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Laboratory-scale model of carbon dioxide deposition for soil stabilisation

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ABSTRACT

Olivine sand is a natural mineral, which, when added to soil, can improve the soil's mechanical properties while also sequester carbon dioxide (CO_2) from the surrounding environment. The originality of this paper stems from the novel two-stage approach. In the first stage, natural carbonation of olivine and carbonation of olivine treated soil under different CO_2 pressures and times were investigated. In this stage, the unconfined compression test was used as a tool to evaluate the strength performance. In the second stage, details of the installation and performance of carbonated olivine columns using a laboratory-scale model were investigated. In this respect, olivine was mixed with the natural soil using the auger and the columns were then carbonated with gaseous CO_2. The unconfined compressive strengths of soil in the first stage increased by up to 120% compared to those of the natural untreated soil. The strength development was found to be proportional to the CO_2 pressure and carbonation period. Microstructural analyses indicated the presence of magnesite on the surface of carbonated olivine-treated soil, demonstrating that modified physical properties provided a stronger and stiffer matrix. The performance of the carbonated olivine-soil columns, in terms of ultimate bearing capacity, showed that the carbonation procedure occurred rapidly and yielded a bearing capacity value of 120 kPa. Results of this study are of significance to the construction industry as the feasibility of carbonated olivine for strengthening and stabilizing soil is validated. Its applicability lies in a range of different geotechnical applications whilst also mitigates the global warming through the sequestration of CO_2.

1. Introduction

Traditional calcium-based binders (i.e. lime and cement) are frequently used as additives for ground improvement (Sariosseiri and Muhunthan, 2009; Celaya et al., 2011; Horpibulsuk et al., 2011; Dash and Hussain, 2012). However, their production involves the emission of carbon dioxide (CO_2). This is a contributing factor to the significant global warming expected in future decades. The production of these traditional binders, i.e. lime and cement, typically produces 600–700 kg and 800–900 kg of CO_2 per tonne, respectively. This is because they require energy (both fuel and electricity) and the production process releases CO_2. For instance, the cement industry alone accounts for around 5% of global CO_2 emissions (Feely et al., 2004; Sabine et al., 2004).

Recent soil stabilisation methods have highlighted the need for full or partial replacement of the traditional binders with cleaner and more sustainable materials (e.g. reactive magnesia, zeolite, fly ash, rice husk ash, cement kiln dust, calcium carbide residue, palm oil fuel ash, ground granulated blast furnace slag) (Rahman, 1987; Yin et al., 2008; Horpibulsuk et al., 2009; Jegandan et al., 2010; Kroehong et al., 2011; Kampala and Horpibulsuk, 2013; Yi et al., 2014; Pourakbar et al., 2015a, b). Most of these methods have focused on using alternative materials to replace traditional cementitious binders and reduce greenhouse gas emissions.

Another promising technique to reduce the industry's greenhouse gas emissions involves capturing the CO_2 to make other products such as carbonates or bicarbonates. It is an attractive sequestration method for the permanent and safe storage of CO_2. Mineral carbonation is a process whereby CO_2 reacts chemically with calcium- and/or magnesium-containing minerals to form stable carbonate phases (Oelkers et al., 2008).

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Serpentine, olivine, wollastonite, steel slag, Estonian oil shale ash, and Mg-oxides have been widely proposed for direct carbonation by several researchers (Maroto-Valer et al., 2004; Hänchen et al., 2006; Liu, 2006; Hangx and Spiers, 2009; Velbel, 2009; Bernal et al., 2010; Rudge et al., 2010; Sissmann et al., 2013; Yi et al., 2013a–c; Song et al., 2014). Accordingly, the recent studies demonstrated that the in situ carbonation can be achieved by injection of CO_2 through a perforated pipe installed in the ground (Andreani et al., 2009; Yi et al., 2013a–c; Cai et al., 2015).

Amongst a wide variety of natural materials for the mineral carbonation process, olivine Mg_2SiO_4 is one of the most promising candidates (Kelemen and Hirth, 2012; Olsson et al., 2012; Saldi et al., 2013). Olivine is widely distributed around the world: Egypt, Myanmar, South Africa, Russia, Pakistan, Norway, Sweden, France, Brazil, Germany, Mexico, Australia, China and also the USA. Geological survey of Malaysia shows that there is a large number of volcanic rocks of the andesite-dacite-basalt in Tawau Mountains in Sabah which is the main source of olivine (Tahir et al., 2010).

The CO_2 from the atmosphere dissolves in available water forming carbonic acid with a pH value around 5.6. Then, the dissolution of olivine in the carbonated water occurs, and finally a stable magnesium carbonate ($MgCO_3$) and silica are precipitated. The carbonated products may have potential to bond soil particles together (Fasihnikoutalab et al., 2015). The carbonation reaction of Mg-rich olivine can be described by

$$\begin{aligned} &Mg_2SiO_4 + 4CO_2 + 2H_2O \rightarrow 2Mg^{2+} + SiO_2 \\ &+ 4HCO_3^- \leftrightarrow 2MgCO_3 + SiO_2 + 2CO_2 + 2H_2O \end{aligned} \tag{1}$$

The aim of this study is to investigate the strength performance of carbonated olivine treated soil at different carbonation pressures and times. In this stage, unconfined compression test was used as a tool to evaluate the performance. Moreover, the bearing capacity of treated soil using carbonated olivine columns was determined by a laboratory-scale set of experiments using specific pressure and time. Microstructural analysis of olivine under the natural condition and carbonated olivine treated soil was also traced.

2. Materials and method

2.1. Properties of materials used

2.1.1. Soil properties

A clayey soil composed of 10% sand, 60% silt and 30% clay was used. The optimum water content and maximum dry density of natural soil were 23.3% and 1.58 g/cm^3, respectively. Liquid and plastic limits were determined to be 54% and 30%, respectively. Based on the unified soil classification system (USCS), the soil was classified as highly plastic (CH). Table 1 shows the engineering properties of soil and 15% olivine sand treated soil. Fig. 1 clarifies the particle size distribution of soil.

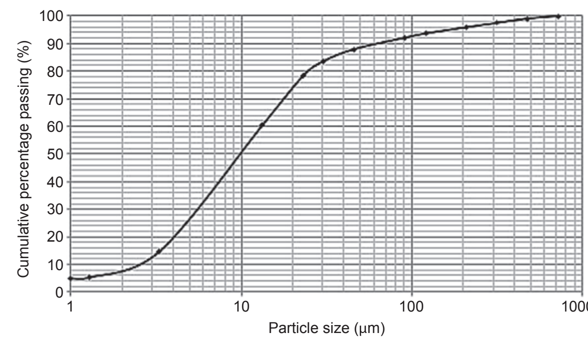


Fig. 1. The particle size distribution curve of soil.

2.1.2. Olivine properties

The olivine in this investigation was supplied by the Maha Chemicals Company of Malaysia. Fig. 2 shows the particle size distribution obtained from a sample of the olivine supplied by the supplier. A laser diffraction particle size analyser (Mastersizer 2000E, ver. 5.52) was used to determine the specific surface area of olivine. The specific surface area and D_{50} (mean particle size) of the olivine sand were 6.07 m^2/g and 2.24 μm, respectively. Table 2 shows the chemical composition of olivine sand according to the supplier in this study.

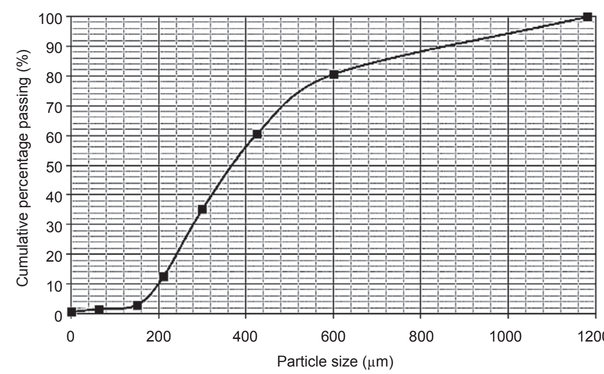


Fig. 2. The particle size distribution curve of olivine.

Analysis using a Jeol 6480 LV scanning electron microscope (SEM) revealed particles with sizes ranging from approximately 500 μm down to sub-micrometre sized particles (see Fig. 3). Moreover, the crystalline phase composition of the olivine was determined by X-ray diffraction (XRD). Data were collected over the 2θ range from 3° to 50°. The relative intensities of the peaks at the critical 2θ angles can be seen in Fig. 4. Analysis of the XRD data confirmed the presence of peaks at 2θ values of 16.55°, 24.8°, 25.52°, 32°, 36.5°, 39.36°, and 44.58° which are in agreement with those reported by Swanson and Tatge (1951) for forsterite, the Mg-rich form of olivine.

Table 2
Chemical composition of olivine sand (%).

MgO	SiO_2	Al_2O_3	Fe_2O_3	CaO	Loss on ignition
48.28	40.32	1.37	8.9	–	1.13

Table 1
Engineering properties of soil and 15% olivine sand treated soil.

Optimum water content (%)	Optimum water content of soil + 15% olivine sand (%)	Plastic limit (%)	Liquid limit (%)	Plasticity index (%)	Specific gravity	Maximum dry density (g/cm^3)	Maximum dry density of soil + 15% olivine sand (g/cm^3)	Particle content (%)		
								Sand	Silt	Clay
23.3	17.4	30	54	24	2.65	1.58	1.719	10	60	30

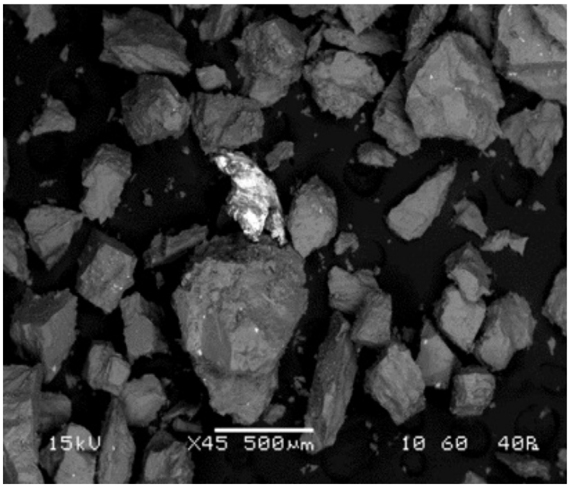


Fig. 3. SEM graph showing olivine sand particles with sizes ranging from a few micrometres to approximately 1 mm.

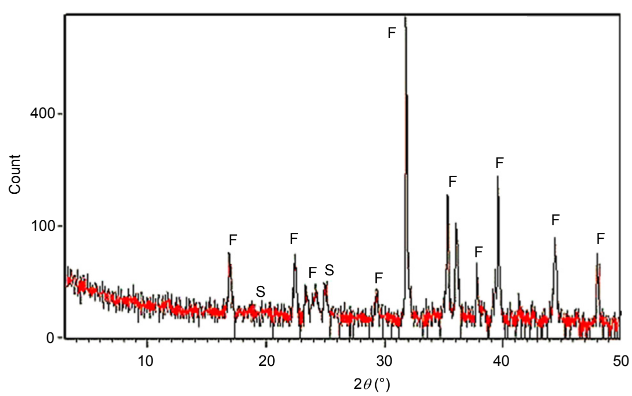


Fig. 4. X-ray powder diffraction pattern of olivine (F: forsterite, S: serpentine).

2.2. Laboratory tests

2.2.1. Raman spectroscopy and field emission scanning electron microscope of carbonated olivine

The natural carbonation of olivine exposed to the Malaysia atmosphere for 6 months was investigated using field emission scanning electron microscope (FESEM) and Raman spectroscopy. A Renishaw's inVia Raman spectrometer with a 25 mW wavelength stabilised diode laser of wavelength 785 nm operating at 1% laser power was used to characterise the olivine. In order to obtain sharp images at high magnification, the olivine particles were first coated with a 10 nm thick layer of chromium, using a Quorum Q150TS turbo-pumped sputter coater. Two samples were prepared for viewing: one with the olivine as received and one with a sample that had been prepared with freshly exposed fracture surfaces. The olivine particles were then viewed in a JSM-6301F FESEM.

2.2.2. Cell setup for carbonation of soil-olivine sample

The olivine was applied to the soil at a concentration of 15% of weight. Directly after mixing, samples were placed in a cylindrical mould (50 mm in diameter by 100 mm in height), applying consistent moderate compaction in three equal layers at the maximum dry density and optimum moisture content of the mixture (see Table 1). Compaction was conducted manually by a 45 mm diameter steel rod to eliminate air pockets, so as to improve the homogeneity of the samples. A cell was used to permeate pressurised CO_2 into the olivine treated soil (Fig. 5). Samples were subjected to a confining pressure of 400 kPa, followed by upward permeation of the CO_2. The outflow tube was placed underwater in order to detect whether or not the samples were fully saturated with CO_2. After a few minutes, the outflow tap was closed while keeping the inlet open. Since the CO_2 mineralization of olivine is significantly influenced by CO_2 exposure time and pressure to olivine (Kwon et al., 2011), the samples were carbonated at various CO_2 pressures of 100 kPa and 200 kPa at various carbonation periods of 7 h, 48 h and 168 h.

In order to assess the effect of carbonated olivine on the strength performance of the samples, the UCS was determined for soil in the 'as-received' condition in addition to olivine treated sample. These tests were performed at different carbonation pressures and periods as specified earlier according to BS 1377-7 (1990).

2.2.3. Microstructural tests

The stabilised soil after carbonation using the cell setup was characterised using JSM 5700 FESEM and SEM to determine the structural information. Elemental information was gained from energy-dispersive X-ray (EDX) analysis and details of the crystalline phases presented were determined from XRD studies. These techniques together provided important information used to investigate the effect of olivine carbonation on the stability of treated soil.

2.2.4. Laboratory-scale carbonated olivine-soil column

A similar methodology to that of in-situ soil by using an auger, followed by its carbonation with CO_2 through a perforated pipe installed in the ground (Yi et al., 2013a–c; Cai et al., 2015), was used for the laboratory-scale model. In this model of ground, a rigid box having dimensions of 800 mm in width, 400 mm in length and 320 mm in height was used (Fig. 6). Three sides of the box were made of steel and the front side of the box had a removable 20 mm thick rigid Perspex panel to allow real-time visualisation of soil

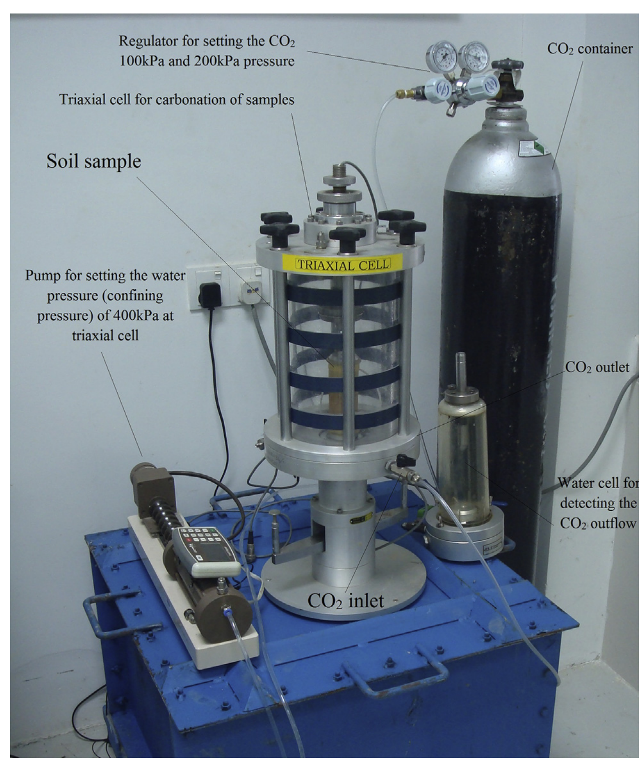


Fig. 5. Cell setup for carbonating olivine treated soil.

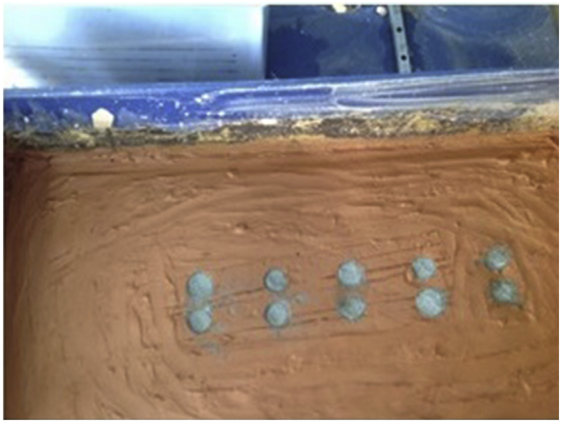


Fig. 6. Physical model of soil-olivine column.

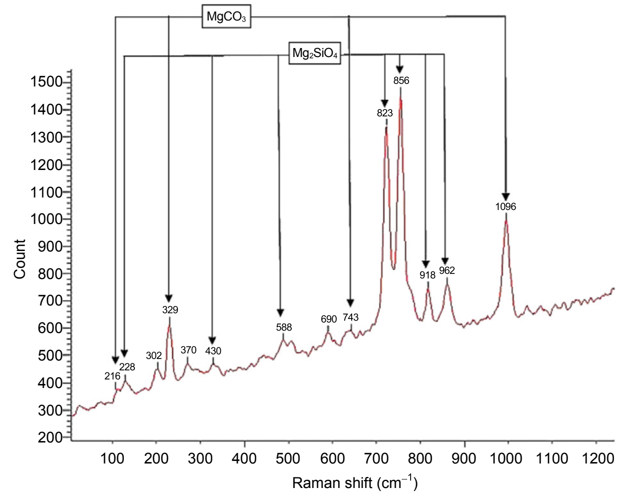


Fig. 8. Raman spectrum obtained from the surface of an olivine particle exposed to the atmosphere for 6 months. Characteristic peaks are shown for the olivine and surface formed magnesium carbonate.

movement during testing. To prepare the scaled model of the ground, the natural soil (clay with high plasticity) was first air-dried under laboratory conditions. While waiting for the soil to be air-dried, the water was carefully poured into a mixing drum. Subsequently, the air-dried soil was slowly and carefully added and allowed to submerge under the water before mixing was started to avoid spreading of dust. The slurry, at just over 2 times the liquid limit, was made up to produce a homogeneous sample as suggested by Sheeran and Krizek (1971). The slurry was mixed for 10 min and then placed inside the steel box in 3 equal layers. The soil was then consolidated for 2 d.

For column preparation, the soil-olivine columns were prepared inside a set of acrylic tube (23.5 mm in diameter and 200 mm in length) by mixing the natural soil and 15% olivine with distilled water. Totally 10 columns of soil-olivine were prepared through the mixing procedure. Provision was also made to produce 2 samples for unconfined compression tests in order to determine the strength and consistency of column mixture. After mixing procedure, 12-d curing periods were adopted. For this purpose, after extrusion from acrylic tube, all prefabricated columns were closely wrapped in polythene covers to avoid water loss and cured in a laboratory ambient environment.

To excavate the soil, a thin-wall brass tube with an outside diameter of 25 mm was pushed slowly into the model ground at the predetermined locations identified by the template. A template with predetermined drilled column location in a 5 mm thick aluminium pattern was used to act as a guide to remove the soil by a laboratory auger for column installation. Note that the improvement area ratio was 11.9%. The soil-olivine columns were slowly installed in the process.

The soil-olivine columns were then carbonated with gaseous CO_2. A plastic pipe with a diameter of 20 mm was inserted into the columns centre. The top inlet of the tube was connected to the CO_2 container, and CO_2 was pumped at 50 kPa pressure into the columns for 1 h (Fig. 7).

3. Results and discussion

3.1. Carbonation of olivine under atmospheric condition

Carbonation of olivine sand under atmospheric condition was confirmed empirically using Raman spectroscopy, as shown in Fig. 8. The main peaks for olivine (forsterite) corresponding to the SiO_4 as reported by Cnopnras (1991) were identified. A peak at 228 cm^{-1} corresponded to the rotational and translational lattice vibration mode. Peaks at 430 cm^{-1} and 588 cm^{-1} corresponded to the ν_2 and ν_4 bending modes, peaks at 823 cm^{-1} and 856 cm^{-1} corresponded to the ν_1+ν_3 stretching modes, and peaks at 918 cm^{-1} and 962 cm^{-1} corresponded to the ν_3 stretching modes. Magnesite

Fig. 7. Physical model of soil-olivine column during carbonation.

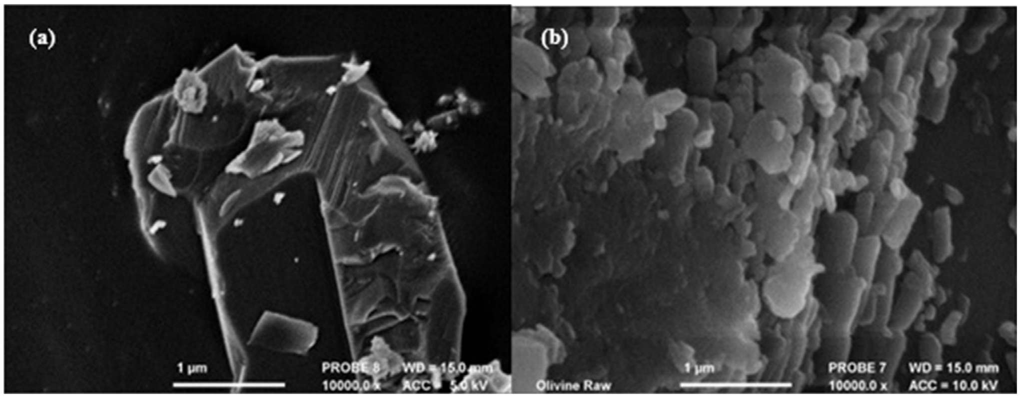


Fig. 9. FESEM graphs of olivine. (a) Morphology of a freshly produced fracture surface on an olivine particle prior to reaction with atmospheric CO_2. (b) Magnesite crystal growth on the surface of an olivine particle exposed to the atmosphere for 6 months.

($MgCO_3$) was identified by peaks at wave numbers of 216 cm^{-1} and 329 cm^{-1} which corresponded to lattice mode vibrations. In-plane symmetric bend vibrations were observed through a peak at 743 cm^{-1} and internal symmetric stretch vibrations of the carbonate ion CO_3^{2-} were observed at 1096 cm^{-1} (Walker and Pavía, 2010), proving that carbonation has taken place.

The presence of magnesite on the surface of the olivine particles was further confirmed by FESEM. Fig. 9a shows the smooth and uncarbonated surface of fresh ground olivine particles. In comparison, Fig. 9b shows magnesite crystal growths on the olivine particle surfaces indicating carbonation.

3.2. Unconfined compressive strength of carbonated soil-olivine samples

Fig. 10 indicates the stress–strain behaviours of soil and soil-olivine samples before and after carbonation at different pressures and carbonation periods. As seen from the figure, untreated samples showed a ductile behaviour with unconfined compressive strength (UCS) value of 100 kPa at a failure strain of 1.8%. The UCS levels of the soil sample increased when the samples were subjected to a confining pressure of 400 kPa for different curing times. However, when the olivine-soil sample was subjected to pressurised CO_2, carbonation of olivine-treated soil led to a stronger and stiffer matrix. The study showed that the rate of strength development was dependent on the CO_2 pressure and carbonation period. At the same period of carbonation, a rapid strength increase can be seen when the CO_2 pressure increased from 100 kPa to 200 kPa, confirming that carbon mineralization is related to CO_2 pressure.

The higher CO_2 pressure increased the acidity of the medium and more Mg^{2+} was released for carbonation (De Silva et al., 2009; Mo and Panesar, 2012). As CO_2 is chemically fixed into the olivine matrix, various products including nesquehonite ($MgCO_3 \cdot 3H_2O$), hydromagnesite ($(Mg)_5(CO_3)_4(OH)_2 \cdot 4H_2O$), and dypingite ($(Mg)_5(CO_3)_4(OH)_2 \cdot 5H_2O$) are formed between soil particles with the effect of bonding them together. Fig. 10 highlights the significant contribution of carbonation to strength where the UCS increased to 1274 kPa under a CO_2 pressure of 200 kPa after 168 h of curing. This supports the strategy that when olivine is used as a soil stabiliser in geotechnical applications, it can benefit the environment through sequestrating CO_2 whilst also strengthening the soil.

Fig. 11 demonstrates the difference between Young's moduli ($E_{50\%}$) of carbonated olivine treated soil at various carbonation pressures of 100 kPa and 200 kPa and carbonation periods of 7 h, 48 h, and 168 h. Note that the Young's modulus was calculated by

$$E_{50\%} = UCS_{50\%}/\varepsilon_{50\%} \tag{2}$$

where $\varepsilon_{50\%}$ is the half of axial strain at the half of UCS failure point. Fig. 11 highlights a general trend of increasing stiffness

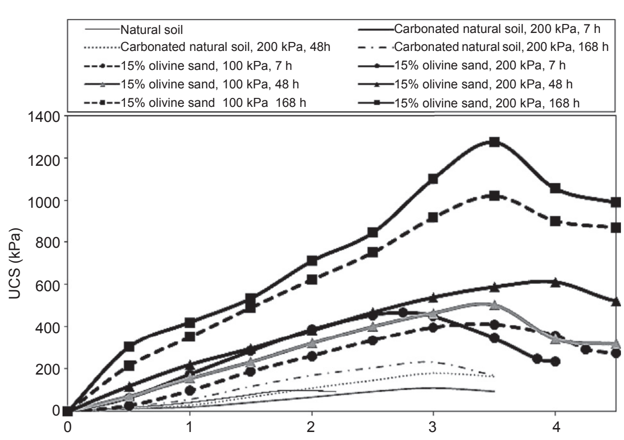


Fig. 10. UCSs of natural soil, carbonated natural soil at 200 kPa CO_2 pressure after 7 h, 48 h and 168 h and 15% olivine treated soil at 100 kPa and 200 kPa CO_2 pressures and 7 h, 48 h and 168 h carbonation times.

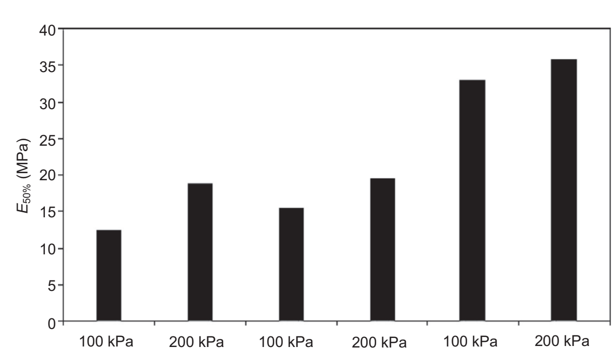


Fig. 11. The Young's modulus of carbonated 15% olivine treated soil at different CO_2 pressures (100 kPa and 200 kPa) and different carbonation times (7 h, 48 h and 168 h).

Table 3
The summary of peak strength and $E_{50\%}$ of carbonated 15% olivine sand treated soil at different carbonation pressures and times.

Samples	Carbonation pressure (kPa)	Carbonation time (h)	UCS (kPa)	$E_{50\%}$ (MPa)
Soil			100.03	
Carbonated untreated soil	200	7	110.68	
		48	180.09	
		168	231.78	
15% Olivine sand treated soil	100	7	410.45	12.5
	100	48	503.04	15.5
	100	168	1020.11	33
	200	7	466.35	18.9
	200	48	612.23	19.6
	200	168	1274.01	35.8

with increasing carbon pressure and carbonation period. The summary of peak strength and $E_{50\%}$ of carbonated 15% olivine sand treated soil at different carbonation pressures and times is presented in Table 3. The greatest Young's modulus of 35.8 MPa corresponded to the 15% olivine treated soil exposed to the highest CO_2 pressure (i.e. 200 kPa) and the longest carbonation period (i.e. 168 h).

The results demonstrate how olivine could be used as a sustainable and promising soil stabiliser with potential to be utilized for soil strength enhancement purposes. Of particular importance is the fact that the soil was stabilised without compromising the environment. This is the advantage of olivine compared to the cement and lime. Cement and lime industries contribute significant global anthropogenic CO_2 emissions as a consequence of the calcination stage of the production process (Worrell et al., 2001; Ke et al., 2013). The associated release of CO_2 into the atmosphere has raised some environmental concerns due to its activity as a greenhouse gas, while olivine with the high naturally occurring MgO content (around 50%) makes it a sustainable product for soil improvement. The use of olivine as a soil stabiliser is considered as a proactive strategy in soil stabilisation. Through the application of olivine, the impacts and risks to the environment are minimised. Moreover, the olivine could be the key to addressing climate change issue by reducing the CO_2 levels of the atmosphere through the CO_2 sequestration process; therefore, olivine is an eco-effective method for soil improvement.

3.3. Microstructural analysis of olivine treated soil following carbonation

3.3.1. XRD analysis of carbonated olivine treated soil

Fig. 12 shows the XRD diffractograms of the carbonated 15% olivine stabilised soil at CO_2 pressure of 200 kPa after carbonation periods of 7 h, 14 h and 168 h, respectively. These diffractograms confirm that the improvement of soil strength was based on the carbonation of olivine in the soil through the hydration and crystallization of MgO. Evidently some brucite peaks are identified as a result of olivine reacting with water. However, by increasing the carbonation periods of 15% olivine stabilised soil from 7 h to 168 h, the weak peak of MgO (Mg) with weak peaks of brucite (B) and serpentine (S) suggests the rapid hydration and carbonation of the MgO, resulting in the formation of nesquehonite (N) and dypingite-hydromagnesite (D/H). Also, according to Eq. (1), the carbonation of olivine not only results in the production of $MgCO_3$, but the XRD diffractograms also suggest it can break the chemical bond between MgO and SiO thus producing quartz (see Fig. 11).

3.3.2. SEM and EDX analyses of carbonated olivine treated soil

Fig. 13 shows the SEM images of the natural soil, 15% olivine stabilised soil after carbonation at CO_2 pressure of 200 kPa and carbonation times of 7 h, 48 h and 168 h, respectively. The figure reveals the formation of intergrowth carbonates $MgCO_3$ agglomerates with edges manifested in soil structure surfaces. According to previous studies, the formation of $MgCO_3$ in soil was responsible for blocking capillary pores and bonding the soil particles together (Yi et al., 2013a–c). The morphology of carbonated olivine treated soil shows a denser structure compared with the untreated soil. It is evident that by increasing the carbonation period from 7 h to 168 h, the soil structure becomes denser (see Fig. 13b–d) and this can be attributed to the substantial carbonation of olivine. Results are in agreement with previous studies reporting that increases in soil strength as a result of the volume increase are observed when magnesium oxide undergoes carbonation forming crystalline $MgCO_3$ (Yi et al., 2013a–c).

Fig. 14a shows the surface analysed and the location of an EDX spot analysis and Fig. 14b is the spectrum analysis of point 1 of carbonated soil containing 15% olivine. The crystalline phase contained Mg, C, Al, Si, Fe and O. The EDX elemental analysis of point 1 reveals the presence of both Mg and C in the round and edge shaped carbonates, suggesting that the carbonates were $MgCO_3$. This is supported by the results shown in Fig. 9b.

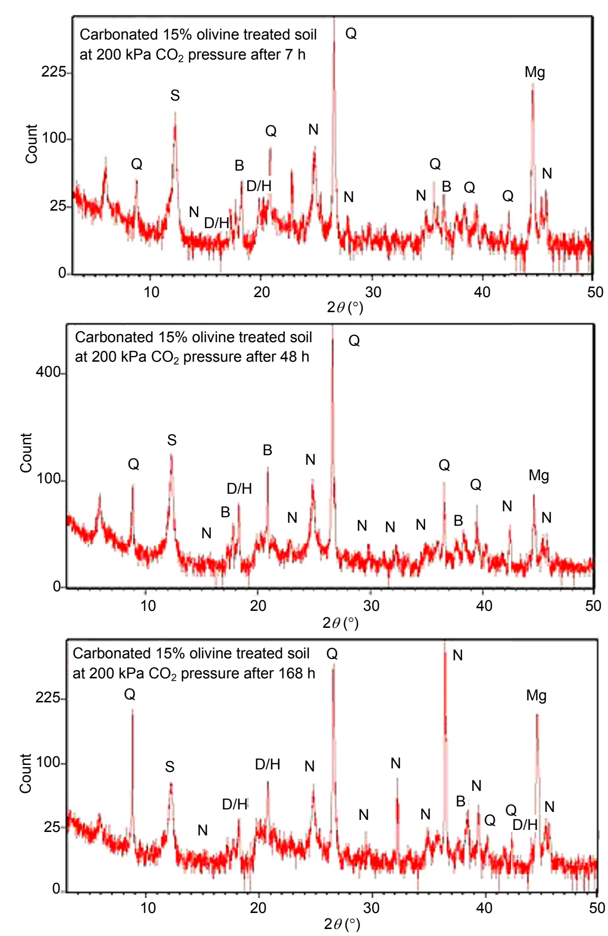


Fig. 12. XRD diffractograms of carbonated 15% olivine treated soil at CO_2 pressure of 200 kPa after carbonation periods of 7 h, 48 h and 168 h, respectively (B: brucite; D/H: dypingite-hydromagnesite; Mg: Magnesium; N: nesquehonite; Q: quartz; S: serpentine).

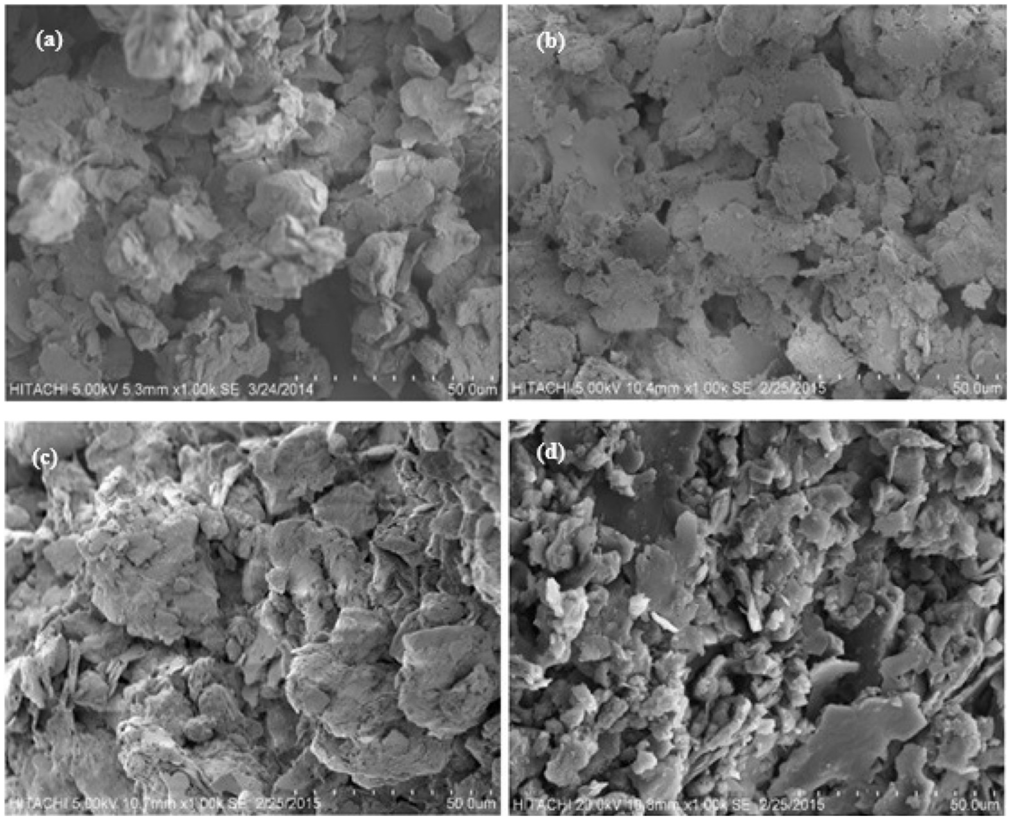
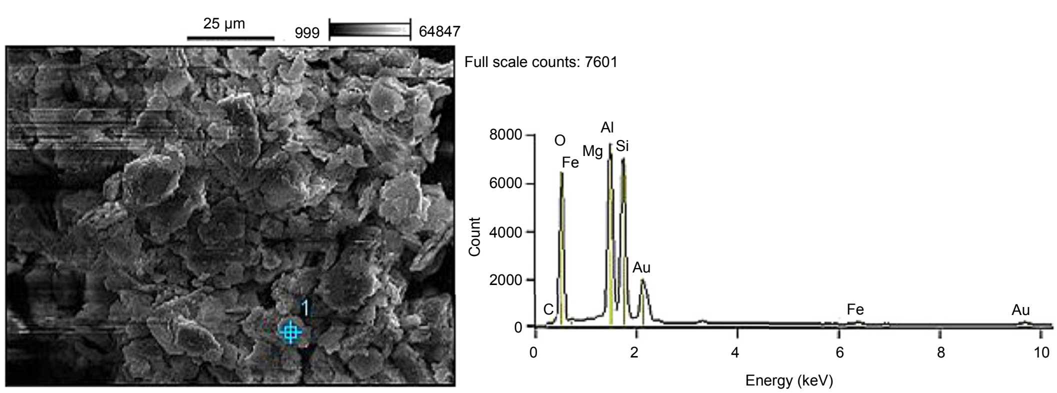


Fig. 13. SEM images of (a) untreated soil and (b) 15% carbonated olivine treated soil after carbonation time of 7 h at 200 kPa, (c) 15% carbonated olivine treated soil after carbonation time of 48 h at 200 kPa, and (d) 15% carbonated olivine treated soil after carbonation time of 168 h at 200 kPa.



(a) (b)

Fig. 14. EDX analysis of carbonated olivine treated soil at CO_2 pressure of 200 kPa and after 168 h carbonation. (a) Point 1 and (b) its spectrum analysis.

Furthermore, according to EDX analysis, the presence of Si in point 1 shows production of quartz in the soil structure according to Eq. (1). The microstructural analysis confirmed the role of $MgCO_3$ in increasing the strength of soil attributed to carbonation.

3.4. Ultimate bearing capacity value

It can be seen in Fig. 15 that the vertical load in untreated case increases with the increase of settlement at first and reaches a plateau at about 0.09 of displacement/footing width. In this respect, the ultimate bearing capacity value of 40 kPa was achieved.

As shown in this figure, compared to untreated model ground, the ultimate bearing capacity value increased sharply in the treated case. In the treated case, the ultimate bearing capacity value of 110 kPa was attained. This sharp increase in bearing capacity of model ground is attributed to the use of carbonated soil-olivine columns. Moreover, during the carbonation procedure of the treated columns and according to previous results, the carbonation of olivine treated soil increases the strength of soil because of the produced $MgCO_3$ and also increases the bearing capacity value of carbonated soil-olivine columns.

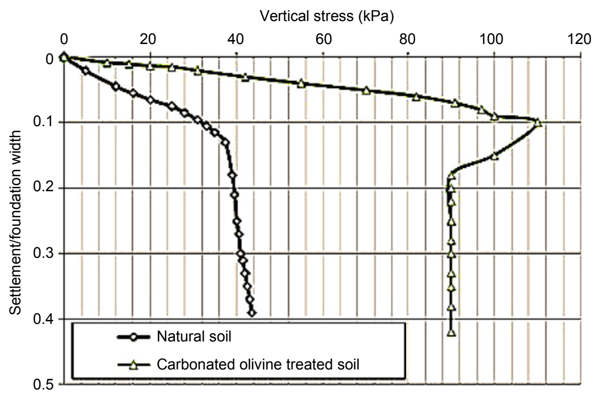


Fig. 15. Ultimate bearing capacity of ground treated by carbonated olivine/soil mixture.

4. Conclusions

This study has demonstrated that following six months exposure to ambient Malaysian atmospheric conditions, magnesite is formed on the surface of olivine. The presence of magnesite was confirmed using FESEM and Raman spectroscopy.

Olivine additions to soil of 15% by mass were found to be an effective level for soil stabilisation. A cell setup was used to permeate pressurised gaseous CO_2, for varying carbonation periods, into the soil revealing the effect of olivine on compressive strength. The UCS of soil was found to increase to 1.274 MPa, which is roughly 12 times greater than untreated soil, when a CO_2 pressure of 200 kPa was applied for a period of 168 h.

The results confirm that utilization of olivine in soil stabilisation is a viable method to improve the bearing capacity. Additionally, it can be adopted to tackle climate change by CO_2 sequestration and strengthen soil for more demanding geotechnical applications.

Conflict of interest

We wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

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